



Fabrication of p-NiO/n-TiO₂ nano-tube arrays photoelectrode and its enhanced photocatalytic performance for degradation of 4-chlorophenol



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ABSTRACT

In the study, we report that p-type NiO (p-NiO) nanoparticles were successfully decorated onto the surface of n-type TiO₂ nano-tube arrays (n-TiO₂ NTAs) photoelectrode by anodization process, followed by electrodeposition strategy in the presence of NiSO₄ electrolyte. After then, the samples were studied by scanning electron microscope (SEM), X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). In addition, the optical and photoelectrochemical (PECH) properties of p-NiO/n-TiO₂ NTAs were recorded through ultraviolet–visible diffuse reflectance spectroscopy (UV–vis DRS), open circuit potential (OCP) and electrochemical impedance spectroscopy (EIS) curves, respectively. Furthermore, We evaluated the photocatalytic (PC) degradation of 4-chlorophenol and the enhanced PC mechanism was also discussed. Results suggest that p-NiO/n-TiO₂ NTAs photoelectrode exhibited higher PC activities than that of pristine TiO₂ NTAs owing to its higher photogenerated electrons–holes (e/h⁺) pairs separation efficiency and light absorbance. As a consequence, the p-NiO/n-TiO₂ NTAs photoelectrode could be applied in degradation of organic pollutants and wastewater purification.

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1. Introduction

Chlorophenols (CPs), as a series of recalcitrant and high-priority pollutants, are extensively used in the manufacture of many aspects such as herbicides, insecticides, disinfectants and dyes [1–3]. They are also frequently produced during industrial production and pollutant disposal. Moreover, 4-chlorophenols (4-CP), as one of commonly CPs, are hardly biodegradable and difficult to remove from the environment by conventional treatment technologies because of their potent acute toxicity and persistence, and the release of which could have great impact on the environment and human health. There is thereby an urgent need but it is still a significant challenge to find a way to solve this problem [4–7]. Photocatalytic (PC) oxidation has been recognized as

promising strategies for degradation of organic contaminants. Most of PC oxidation, in particular TiO₂ PC oxidation, was available technology and attracted abundant attentions due to their prospective applications in pollutants elimination, air purification, inactivation of microbes and photoconversion of CO₂ in recent years [8–11]. However, it is vital defects that TiO₂ nanomaterials possess wide band gap (3–3.2 eV) and high recombination rate of photogenerated electrons–holes (e/h⁺) pairs [12–15]. The two major factors significantly restrict quantum yield of photocatalytic process and necessity to use of irradiation, so that its PC activity could be greatly lowered for degradation of contaminants. In order to solve these problems, many different strategies were proposed, such as transition metal cations doping [16], nonmetals doping [17], dye sensitization [18], inorganic combination [19] and surface modification with precious metals and lower band gap semiconductors [20,21].

NiO, as typical p-type semiconductor [22], possesses high holes mobility, high p-type concentration and low cost production. It is generally accepted that the combination of both p-type and

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n-type semiconductor materials could form p–n junctions in contact surface due to carriers diffusive movement. Also, the p–n junctions could further promote the effective electrons–holes (e/h^+) pairs separation and enhance the visible light absorption [23,24]. Therefore, p–NiO was also expected to be a prospective candidate on contacting n–TiO₂ due to an additive pathway associated with the formation of an inner electrical field [25–27]. According to previous reports [28], NiO/TiO₂ composites with different nanostructures such as NiO/TiO₂ nanoparticles and NiO nanoparticles decorated TiO₂ nanobelt/nanotube have been explored through deposition of NiO onto the surface of TiO₂ or direct mixing of both components. However, the separation of traditional NiO/TiO₂ powder is not extremely easy, and it is difficult to reuse from solution system in wastewater purification field. Thus, the direct coating of NiO nanoparticles onto the surface of TiO₂ NTAs photoelectrode prepared by anodization metal Ti foil to form nanocomposite are an available strategy to solve this issue, and it is still a challenge to find a novel approach to construct the NiO/TiO₂ composites.

In this study, highly-ordered and well-aligned TiO₂ NTAs were firstly prepared by anodization of Ti film in the fluorinated containing electrolytes. Whereafter, p–NiO nano-particles decorated n–TiO₂ NTAs photoelectrode was constructed through galvanostatic electrodeposition strategy. Subsequently, the microstructure, optical, surface chemical states and photoelectrocatalytic properties of the resulting p–NiO/n–TiO₂ NTAs photoelectrode were studied systematically. Furthermore, the PC performance for degradation of 4–CP would be measured comparing with pristine TiO₂ NTAs. The mechanism of their enhanced PC activities was also proposed. The as-prepared p–NiO/n–TiO₂ NTAs photoelectrode expectantly exhibited excellent PC activity in the degradation of 4–CP solution under UV–visible light irradiation and high charge separation efficiency, suggesting its potential application as effective photocatalysts.

2. Experimental

2.1. Materials

In this study, all chemicals used were analytical grade as received without any further purification. In detail, ammonium

fluoride, glycerol, nickel sulfate, sodium sulfate, 4–chlorophenol (4–CP), terephthalic acid (TA) and other reagents including ethanol, acetone, hydrofluoric acid (36.4%) and nitric acid (70%) were purchased from Sinopharm Chemical Reagent Co. Ltd. Titanium foil (99.5%, 0.5 mm thick) was purchased from Shenzhen Futai metal materials Co. Ltd. Moreover, deionized (DI) water was used throughout during the experiment.

2.2. Fabrication of p–NiO/n–TiO₂ NTAs photoelectrode

According to previous reports by our group, The TiO₂ NTAs photoelectrode was successfully synthesized through anodic oxidation method on the surface of Ti foil using a conventional cathode–anode device [29,30]. Specifically, the Ti foil was automatically polished with rough papers before the anodization, then cleaned by ultrasound in acetone, ethanol and purified water for 10 min separately and dried at room temperature. Afterwards, the Ti foil was brightened by mixed acid (HF:HNO₃:H₂O = 1:4:5 in volumetric ratio) for 30 s to appear a glossy surface and were washed with DI water. Furthermore, the TiO₂ NTAs were developed on titanium foil via anodization in the electrolyte that was composed of 0.5 wt% NH₄F and 60% (v/v) glycerol in water. The reaction was carried out using a traditional two-electrode system with Pt mesh as cathode and cleaned Ti foil as anode. Eventually, the as-prepared samples were calcined in the resistance furnace at 500 °C for 2 h.

In order to construct NiO nano-crystallites decorated TiO₂ NTAs, TiO₂ NTAs photoelectrode, platinum electrodes and SCE electrode as cathode, anode and reference electrode, respectively, the reaction was carried out at 0.8 mA cm^{−2} for 30 min using galvanostatic electrodeposition method to deposit NiO nano-particles on the surface of TiO₂ NTAs photoelectrode. 0.1 mol L^{−1} nickel sulfate that contained 0.1 mol L^{−1} sodium sulfate solution was used as electrolyte. Finally, as-prepared samples were washed repeatedly by absolute ethanol and DI water, followed by annealing at 500 °C for 2 h.

2.3. Characterization of p–NiO/n–TiO₂ NTAs photoelectrode

The apparent morphologies of p–NiO/n–TiO₂ NTAs samples were seen by using a Quanta 200F field emission scanning electron

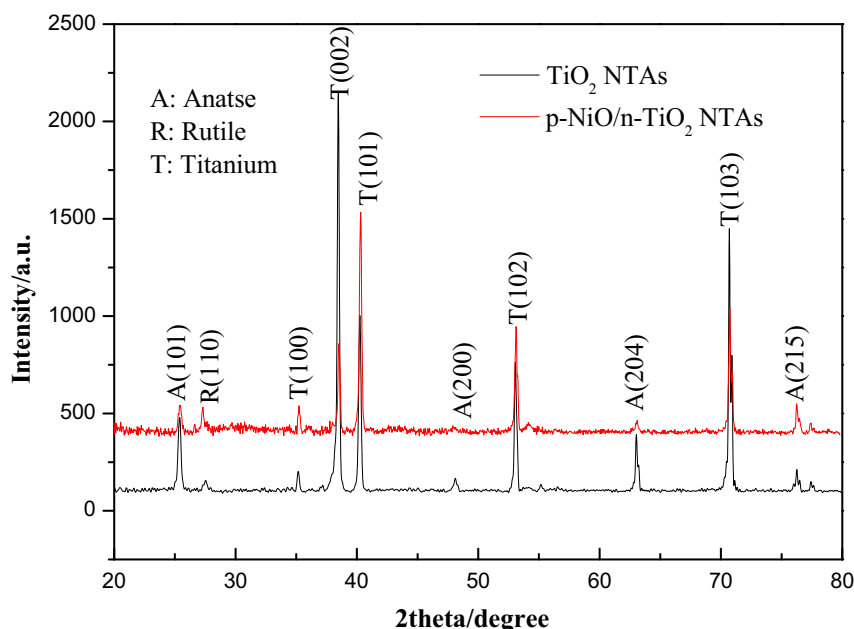


Fig. 1. XRD patterns of pristine TiO₂ NTAs and p–NiO/n–TiO₂ NTAs photoelectrode.

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